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# MULTI-SCALE STRAIN MEASUREMENTS OF A PARTICULAR COMPOSITE MATERIAL

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## ABSTRACT

In this study, the strain fields on two different length scales in a particulate composite material containing hard particles embedded in a rubbery matrix were investigated, using two different techniques. The experimental results were analyzed and are discussed.

## INTRODUCTION

It is well known that, on the microscopic scale, a highly filled polymeric material can be considered an inhomogeneous material. When these materials are stretched, the different sizes and distribution of filled particles, the different crosslink density of polymeric chains, and the variation in bond strength between the particles and the binder can produce highly nonhomogeneous local stress and strain fields. Depending on the magnitude of the local strain and the local stress, damage can be developed in the material, especially near a crack tip region. The damage developed in the material may be in the form of microvoids or microcracks in the binder or dewetting between the binder and the filler particles. The developed damage will not be confined to a specific location; rather, it will diffuse into a relatively large area or zone. The growth of damage in the material may take place by tearing the material or by successive nucleation and coalescence of the microvoids. These damage processes are time dependent and are the main factor responsible for the time sensitivity of strength degradation as well as the fracture behavior of the material. Therefore, in order to gain an advanced understanding of the failure process in these materials, a detailed knowledge of the deformation process as well as damage initiation and evolution mechanisms are required.

In this study, the strain fields for different length scales for a particulate composite material were determined using different experimental techniques. The particulate composite material contains hard particles embedded in a rubbery matrix. The average size of the particle is 200 microns and the volume fraction of particles is 70%. The strain fields near the crack tip in regions of 2mm by 2mm and 20 mm by 20 mm of a sheet specimen under constant strain rate conditions were determined using Digital Image Correlation techniques and Grid methods. The data were analyzed and the results are discussed.

## THE EXPERIMENTS

### *Grid Method*

In this study, the local strain fields near the tip of a crack in the xz plane of a multi-phase material, containing hard particles in a rubbery matrix, subjected to two constant strain rates were determined using the grid method [1]. The specimens were 20.32 cm long, 5.08 cm high, and 0.508 cm thick. Prior to the test, a 25.4 cm crack was cut at the edge of the specimen. Since the specimens were quite soft, a special grating had to be developed from which displacements near the crack tip could be measured. A course grating consisting of squares of 0.2 mm on each side (approximately ½ of the largest size of the hard particle) and which had a thickness of less than $2.5 \times 10^{-2}$ mm was deposited in the neighborhood of the crack tip. The procedure to print the grid on the surface of the specimen was to cover an area of about 5.08 by 5.08 cm with a very thin layer of mixed silicone grease. Then a mesh of 5 lines per millimeter was placed on that area. The grid was pressed gently onto the specimen and the excess grease removed. Then a white colored titanium oxide powder was sprinkled on the specimen surface. When the mesh was removed, a grating showed on the specimen surface.

Prior to testing, the specimens were conditioned at the test temperature (70°F.) for 1 hour and were then tested at constant strain rates of .005 cm/cm/ min. to 0.05 cm/cm/min. normal to the crack plane until the specimens fractured. During the tests, photographs of the grid region were taken at various time intervals and they were used to determine the displacement fields near the crack tip. In addition, a strip chart recorder was used to record the load and time during the test. These data were used to determine the crack growth rate and a "Corresponding" Mode I stress intensity factor. [1]

The determination of the displacements and the strain fields requires digitizing the data from the photographs. When digitizing the data, points located at the intersection of the grating lines are selected close together in regions of expected high gradients and further apart away from these regions. These points in groups of four form quadrilaterals (initially rectangular) and values are read at each of four points in groups and averaged before and after loading. The value of the four points average is located at the centroid of each quadrilateral and the difference between the no-load and loaded values becomes the digitized displacement at that point. This four-point smoothing at a given point will reduce the experimental error of reading the grid and it also reduces the local anomalies that exist in the real deformation of the heterogeneous specimen. The calculated displacement data were stored in a computer and processed to calculate the strains, ($\varepsilon_y$, $\varepsilon_x$, and $\gamma_{xy}$) and to plot the iso-strain contours. In calculating the strains, small strain definitions were used. Therefore, strain contours for strain level greater than 20% should be ignored.

*Digital Image Correlation Techniques*

In this study, edge-cracked sheet specimens were used to determine the strain fields near the crack tip under a constant strain rate (0.067 cm/cm/ min) at room temperature. The specimen was 6.35 cm. long, 3.30 cm. height, and 0.508 cm thick. A 12.7 mm crack was cut at the edge of the specimen with a razor blade.

In this study, California Institute of Technology's testing equipment was used to conduct the constant strain rate tests. It includes a straining stage driven by a stepping motor through a flexible cable, a Nikon microscope, a CCD camera, and a personal computer with a frame grabber unit. The straining stage is mounted on a positioning stage, for which a joystick controller allows the positioning of the straining stage under the Nikon microscope. A detailed description of the testing equipment can be found in Ref. 2.

During the test, two light sources were used and their positions were chosen carefully to minimize the shadows on the specimen surface. The deformation process was monitored with a 200 mm zoom lens that attached to a CCD camera, which captured an area of 24 mm by 18 mm on the specimen surface. The images of the specimen surface were stored at a rate of one image per five seconds, and they were used to determine the strain fields near the crack tip.

In order to check the accuracy of the digital image correlation technique, a specimen of homogeneous silicone rubber without a crack and coated with microscopic speckles was stretched monotonically and uniaxially to a maximum strain of 70% in a sequence of 12 deformation steps of 5.08% strain each. These strains were recorded optically with the aid of a microscope by keeping track of special marks equal to prescribed strain. In addition, the digital image correlation program was used to compute the strain at a given deformation step. A comparison of the prescribed strain, obtained optically, and the computed strain, obtained by digital image correlation technique, revealed that a maximum deviation of 1% occurs at a strain of 40%. This precision is considered to be acceptable for experimental mechanics investigations.

To determine the strain fields near the crack tip, a Large Deformation Image Correlation (LDIC) program, developed by Gonzalez (2) and Vendroux al.et. (3) was used. The LDIC program was developed by modifying a Digital Image Correlation (DIC) program developed by Sutton et. al. (4) for small deformations. The problem in applying DIC to compute strain fields in a large deformation process is the failure of convergence of the DIC algorithm if the strain is larger than 10%. To circumvent this problem, the LDIC takes intermediate images (or steps) of the deformation between the undeformed and the deformed state and then computes the displacements and the displacement gradients for every step of the deformation. The intermediate results are combined to produce the displacements and displacement gradients for the global deformation.

## RESULTS AND DISCUSSION

The results of the strain analysis based on the Digital Image Correlation techniques will be discussed first followed by that based on the Grid methods.

The effect of microstructure on the strain fields near the crack tip is shown by plotting the contours of the iso-intensity principal strain, in Fig.1. The data is presented as maximum principal strain (MPS) rather than the usual components of strain because the crack opening and void formation depend on the local principal strains. In Fig. 1, the crack is shown as the horizontal line with the crack tip at a location of x=0.4 mm and y = 1.25 mm (0.4mm or 1.25mm).

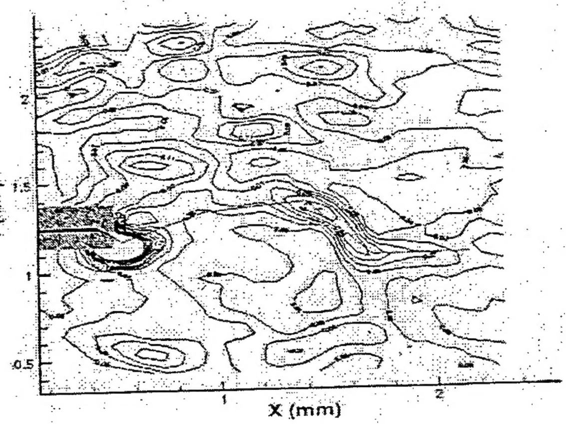


**Figure 1. Maximum Principal Strain At 6% FFS (Far Field Strain) During The Loading Segment Of Test C**

The gray area surrounding the crack is the noise region in which the data cannot be analyzed. This is because the digital image correlation program doesn't converge in places where new geometrical features appear. The cases of crack propagation and void formation are examples in which new features appear in the image. The digital image correlation program doesn't recognize the relationship between the features in the undeformed and the deformed images. Referring back to Fig. 1, it is seen that, starting at the crack tip, there is a broken thin line that goes toward the right of the figure, representing the future crack propagation path. Note that the strain fields are highly nonhomogeneous and high strain regions are localized in the neighborhood of the crack tip. For example, the largest MPS, greater than 20%, is reached about 0.2 mm from the crack tip. There are several other high strain regions located near the crack tip. The average of these high strain regions is about 400 microns in diameter. It is interesting to note that the high strain region located at (1.4 mm and 1.3 mm) is the location where a void will develop as the applied far field strain (FFS) is increased.

A plot of the MPS concentration factor, defined as a ratio of MPS to FFS, along the crack plane as a function of the FFS is shown in Fig.2. From Fig. 2, the curves representing FFS of 1% to 4% fall very close to each other indicating that the distribution of MPS is fairly linear with the FFS. However, when the FFS level increases to 5%, a significant increase in MPS concentration factor occurs in a region between x = 0.7 mm and x = 1.8 mm. This phenomenon is probably due to the separation of the particle and the binder, resulting in a significant increase in MPS. When the FFS is continuously increased, eventually a void is formed in this high strain region and the crack advances by the coalescence of the crack tip with the void.

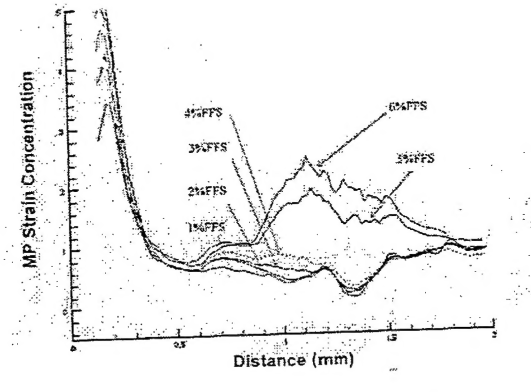


**Figure 2. MPS Along Plane Of The Crack At Various FFS Values. Crack Tip Is At Origin**

In the following paragraphs, we will discuss the results of strain analysis, based on the Grid methods, and the application of a continuum approach to analyze the strain fields near the crack tip.

A typical plot of the normal strain near the crack tip at a far field applied strain of 7.5% is shown in Fig.3. The iso-strain contour lines are not smooth but irregular as a result of the microstructural effect. However, the shape of the contour lines resemble the plastic zone shape observed near the crack tip in metallic materials. This indicates that, on the macro scale, the highly filled polymeric material can be considered a homogeneous continuum, as discussed in the following paragraphs.

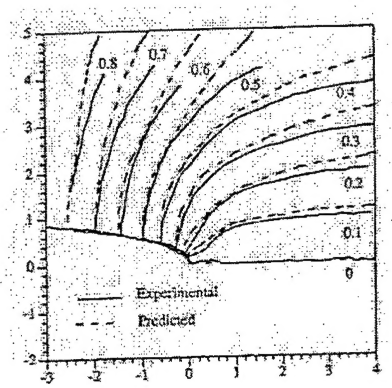


**Figure 3. Contours Of Constant Vertical Displacement (V) In The Cracktip Region Of A Specimen Subjected To A Loading Rate 2.54 mm/min At A Far Field Strain • $_y$ = 7.5%.**

A rather accurate method of analysis can be utilized in order to determine if the fracture behavior can be treated in a continuum fashion. It is well established by Benthem [5] that the order of the singularity used in defining the stress intensity factor (i.e. • . = 1/2 ) takes on a different value when a crack

intersects a boundary at right angles. Using Benthem's three dimensional variables separable eigenfunction expansion of the stresses (• $_{ij}$) and the displacements ($v_i$) near the crack tip at the free surface for a quarter infinite crack intersecting a half space at right angles and linear elastic fracture mechanics (LEFM) as a guide [6] one can construct the following expression for the near tip displacement normal to the crack surface at the crack tip as:

$$v = D_v r^{\lambda u} \quad (1)$$

where r is measured in the direction of v at the surface of the body and • $_v$ is the lowest dominant displacement eigenvalue. (• $_u$ = 1 - |• . | )

Then, in order to account for the extensive stretching of the binder introduced by the presence of the motion of the particles as they move normal to the crack plane ahead of it, under Mode I extension, we eliminate this large strain process zone by introducing a constant value $v_o$ into Equation (1). Thus,

$$v - v_0 = D_v r^{\bullet\, u} \quad (2)$$

As shown in Fig. 4. Once this is done, an average of four tests on the inert propellant resulted in an average value of • $_u$ = 0.657 [6] and was independent of an order of magnitude change in the displacement rate. This compares favorably with Benthem's result for an incompressible material of 0.67. The favorable comparison is despite the fact that the inert propellant may not be incompressible over the full test range. Thus it appears that, by eliminating the large process zone ahead of the crack tip, it is possible to determine • $_u$ rather accurately using a continuum approach. However, it should be borne in mind that, in general, blunting at the surface is not considered to be included in Benthem's Analysis and will increasingly reduce the accuracy of the above procedure [7], [8].

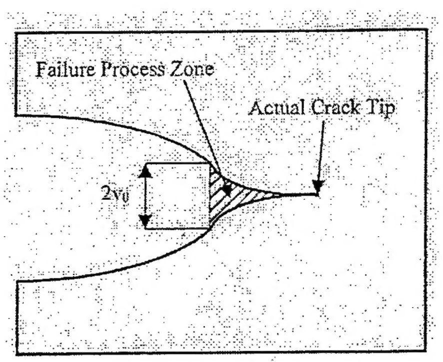


**Figure 4. Use of $v_o$ for Blunted Crack Tip and Location of Actual Crack Tip When Grid is Damaged at Free Surface.**

In addition to determination of the strain field experimentally, a numerical simulation was performed on the sheet specimen, mentioned in the Grid Method section, with appropriate consideration of the symmetry in the problem. The material properties used are obtained from the stress-strain curve shown in Fig. 5. The Young's modulus and the Poisson's ratio for the material are 1.9 MPa and 0.499, respectively. Regular four node quadrilateral elements were used in the computation. The crack tip region contained a focused and refined mesh to adequately capture the crack tip fields.

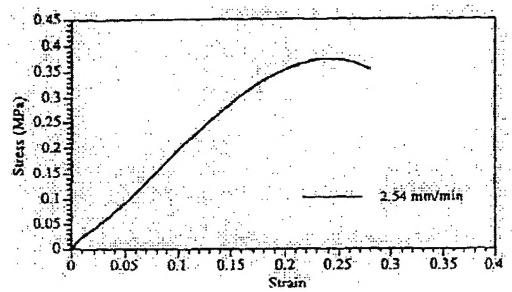


**Figure 5. Stress-Strain Curves for the Particulate Composite at Loading Rates of 2.54 mm/min.**

The results of the finite element analysis are shown in the form of normal displacement contour lines and are superposed on experimental results in Fig. 3. The contours compare reasonably well. Similarly, the normal strain contour lines, obtained from finite element analysis, are superposed on the experimental results in Fig. 6. It is seen that a reasonably good correlation exists between the experimental and the numerical results.

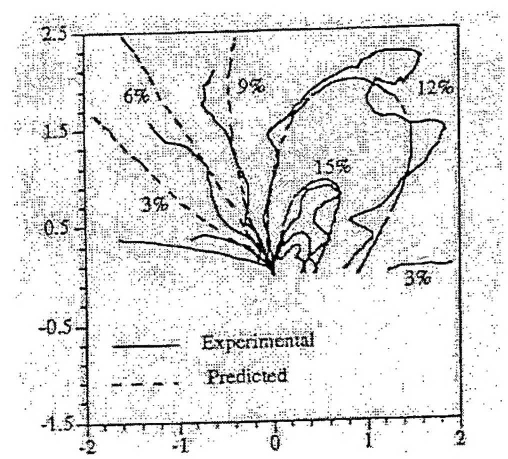


**Figure 6. Finite Element Results for Normal Strain Contours Superimposed Upon Experimental Result.**

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